1321-114 PCT-US

IN THE UNITED STATES RECEIVING OFFICE

Applicants: DeKleine
Serial No.: 10/582,916
Filing Date: March 7, 2007
Based on
International Application No.: PCT/EP2004/052615
Filing Date: October 21, 2004
For: PROCESS TO PREPARE ALKYL PHENYL PHOSPHATES

Mail Stop: AMENDMENT
Commissioner for Patents
PO Box 1450
Alexandria, VA 22313

DECLARATION OF ANDREW M. PIOTROWSKI UNDER 37 CFR §1.132

Sir:

I, ANDREW M. PIOTROWSKI, declare as follows:

1. I am a Scientist for Supresta U.S. LLC, the assignee of the priority patent and the present application.

2. In the Office Action dated September 11, 2008, the Examiner maintained the rejection of Claims 1-10 and 13-21 in the previous Office Action dated November 28, 2007 under 35 U.S.C. §103(a) as allegedly being obvious over United States Patent No. 4,034,023 to Hardy Sr. et al. (hereinafter referred to as "Hardy") in view of United States Patent No. 3,931,367 to Giolito (hereinafter referred to as "Giolito").

3. I submit this declaration under 37 C.F.R. §1.132 in order to demonstrate that it is critical to the present invention that the process is conducted at a temperature *above 35°C* by showing a side-by-side comparison between the claimed process for producing alkylphenyl phosphates comprising the steps of reacting a dichloromonophenyl phosphate and monochlorodiphenyl phosphate with an aliphatic

alcohol, in the presence of a Lewis acid catalyst, in the absence of solvent, at a temperature of above 60 to 200° C, and at a pressure of 0.001 to 1.1 bar absolute pressure (bara) (experiment 3) and Hardy (experiments 1 and 2).

4. Indeed, it has been unexpectedly found that the reaction of dichloromonophenyl phosphate and monochlorodiphenyl phosphate with an aliphatic alcohol, in the presence of a Lewis acid catalyst, in the absence of solvent, at a temperature of *above 60 to 200°C*, and at a pressure of 0.001 to 1.1 bar absolute pressure (bara) produces a product having a higher purity than would have been expected.

5. In order to demonstrate the unexpected results found, three experiments were conducted to provide a direct comparison of the product yield of the process of the present invention and the product yield of the process disclosed in Hardy at the same temperature and concentration.

6. The three experiments conducted show a side-by-side comparison between the claimed process for producing alkylphenyl phosphates comprising the steps of the present invention, which involves reacting a dichloromonophenyl phosphate and monochlorodiphenyl phosphate with an aliphatic alcohol, in the presence of a Lewis acid catalyst, in the absence of solvent, at a temperature of above 60 to 200° C, and at a pressure of 0.001 to 1.1 bar absolute pressure (bara) (experiment 3); and the process described in Hardy (experiments 1 and 2).

7. Experiment 1 was conducted using 2-ethyl hexanol at a temperature of 20°C using the same ratio of alcohol as used in the claimed invention. It is noted that Hardy used 100% excess of alcohol to shorten the reaction time and improve yield. As

shown, in this experiment conducted without excess alcohol, it took more than 30 hours to get a yield of about 84.3 percent. (See, Table 1, hereinbelow).

8. When the same reaction was conducted at a higher temperature (experiment 2), namely 120°C, the reaction was faster but the yield was lower (79.3 percent) due to by-product formation. (See, Table 1, hereinbelow). Further analysis indicates that not only is the yield less pure but the reaction mixture contains significant formation of alkyl chloride which is very undesirable since alkyl chloride is very hard to dispose of chemically. Therefore, experiment 2, which was conducted at a higher temperature and without vacuum, as described in the Hardy reference, produced a less desirable product mixture having more by-products.

9. In stark contrast, experiment 3, which is taken directly from the table in experiment 4 of the specification, and was run at the claimed temperature of 120°C under vacuum, yielded a much higher yield than experiments 1 and 2 following the Hardy teachings. That is, Experiment 3 following the inventive conditions produced over 97 percent product in about 4 hours with much less by-products. (See, Table 1, hereinbelow). Less by-products make the process more cost effective and efficient since less purification is needed and less disposal of harmful chemicals.

10. Accordingly, as shown by the side-by-side comparison of the process run under the Hardy conditions (experiments 1 and 2) and the process run according to the claimed conditions (experiment 3), the temperature used is shown to be critical in producing the claimed process wherein the yield is higher and is done without excess alcohol.

11. In view of the foregoing, and in line with the teachings of Hardy, one

skilled in the art would not have changed the reaction temperature from 20°C as taught by Hardy to above 60°C to 200°C as claimed. Accordingly, carrying out the reaction under the conditions of the present invention unexpectedly produces a more purified product mixture at the same reaction temperature and concentration.

Table 1

#	Example	Reaction Scale (liters)	Reaction Feed Amounts (Kg)	Reaction Conditions				Crude Reaction Product Composition		
			DPCP Mix	2-EH	T (°C)	P (mmHg)	Addition Time (hrs)	Post Addition Reaction (hrs)	2-EHCl (wt%)	2-EH (wt%)
1	1	2	0.75	0.396	20	ambient	1.17	31.25	1.9	2.3
2	2	2	0.75	0.396	120	Ambient	1.75	3	11.7	0.6
3	3	1	0.5	0.264	120	50	1.5	4	0.7	0.4

Table 1 (cont.)

Crude Reaction Product Composition				
TPP (wt%)	2-EHPPh (wt%)	2-EHDPPh (wt%)	DPAP (wt%)	Total Yield
0.4	77.3	7.0		84.3
0.3	75.3	4.5		79.8
3.3	84.1	10		97.4

12. All acts set forth herein took place in the United States.

13. All statements made herein are of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine and imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated: February 11, 2009

Andrew M. Piotrowski

ANDREW M. PIOTROWSKI